# Factors Affecting the Upgrading of Iron Oxide Derived From Fly Ash

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## Introduction

The iron oxide present in fly ash can be removed by magnetic separation methods. The material so obtained has possible useful applications as a heavy media agent in coal preparation and as a raw material for the steel industry. In order to meet the various specifications of the materials now used in these processes, the fly ash-derived iron oxide must be further upgraded. This paper describes some of the problems encountered in upgrading the iron oxide material.

## A. Separation of Iron Oxide from Fly Ash

Iron oxide can be removed from fly ash by either wet or dry magnetic methods. Typical magnetic separations systems which could be applied to this process are shown schematically in Figures 1 and 2.

The wet process (Figure 1) involves pumping a fly ash slurry into a unit containing a magnetic drum. The magnetic iron oxide and associated material are collected from the drum, and the collected material is passed through a similar unit for further refining. The non-magnetic material is transported to a disposal area.

The dry process also uses a rotating magnetic drum. The initial separation produces middlings and tailings fractions, which are discarded, and a crude iron oxide fraction which must be further refined by passing through another magnetic unit.

The iron oxide so obtained is similar in physical and chemical characteristics (Table 1) to commercially available magnetite and therefore can be used, in some cases, as a heavy media agent without further processing.

## B. Processing of Iron Oxide for Use as a Heavy Media Agent

The important factor in the use of fly ash-derived iron oxide as a heavy media agent is the magnetic content of this material. The total magnetics should be as high as possible in order to have high recovery rates at the preparation plant, thereby keeping the consumption of iron oxide per ton of coal at a low rate.

Commercially available magnetite contains 94 percent magnetic material. Samples of iron oxide from fly ash can be produced containing 90 percent magnetics, however this requires further processing of the iron oxide in another magnetic unit, or alternately by crushing the iron oxide to liberate the magnetic material and subsequent magnetic separation to collect the magnetic fraction.

This latter process serves another purpose, namely, that of producing a finer sized material. Heavy media washers of different designs require magnetic material of different size consists for efficient operation. Certain washers can use the fly ash-derived iron oxide as it is produced from the fly ash; others require a finer material. However, further grinding reduces the total magnetics content in the iron oxide. This occurs because non-magnetic materials are present in the iron oxide and are liberated by the grinding process. The reduction in total magnetics can be as much as several percent. Detailed comparison tests are yet to be made on this phase of the project.

## C. Upgrading Iron Oxide for Use as a Raw Material in the Steel Industry

The object of this phase of the program was to refine the iron oxide to obtain a fraction having the highest possible iron content while maintaining a low silica and aluminum content. Considerable attention was therefore focused on the analysis of the iron oxide material. Methods of analysis were developed to determine iron, silicon and aluminum.

Several different refining procedures were attempted.

1. The iron oxide removed from the fly ash was pulverized until 90 percent passed a 325 mesh screen. The crushed material was then washed with water several times; after each washing the iron oxide was allowed to settle and the lighter material removed by decantation. This method did not increase the iron content nor did it decrease the silica content, however the aluminum content was reduced from 6.0 percent to 3.7 percent.

2. A second attempt at purification was attempted using the following method. The iron oxide was pulverized to 98 percent through a 325 mesh screen. The magnetic iron was removed by a dry magnetic method and then processed by specific gravity separations at 2.95 specific gravity. This method also did not improve the iron or silica contents, however it decreased the aluminum to 0.3 percent.

The results of these experiments showed that the iron and silica are intimately combined in such a form that grinding and washing will not make possible a separation. This combination is described as "silicated iron oxide" and has an iron-to-silica ratio of 4.6 to 1.

3. A third attempt at reducing the silica content was attempted. This method involved crushing the original, unprocessed fly ash to 98 percent through a 325 mesh screen, however when the resultant material did not yield any iron oxide when magnetic separations were attempted. The magnetic properties of the iron oxide apparently had disappeared.

These results led to a closer investigations of the physical and chemical characteristics of the fly ash.

## D. Analysis of Iron Oxide for Iron Content

No standard wet analytical method for iron in fly ash was available for use in this work. Initially, the ASTM methods recommended for cement were used in which the iron was extracted from the fly ash by hydrochloric acid. From the color of, and microscopic examination of, the residues, it was suspected that all of the iron was not extracted by hydrochloric acid. Sodium carbonate and caustic fusions were attempted and gave higher results than the hydrochloric acid extraction (Table 2). However, it was again suspected that some of the iron had not been dissolved. Hydrofluoric acid extraction of the samples was then attempted and gave results which were consistently reproducible. The hydrofluoric acid extraction method gave the highest results.

## E. Microscopic Examination

It was believed that the problems with the analyses and processing of the fly ash-derived iron oxide were associated with the silicated iron material. Microscopic examination supported this belief.

Pollen amounts of (a) fly ash, (b) the residue from the magnetic separation process and (c) the magnetic iron oxide were examined and photographed under a microscope at 200 and 400 magnification using transmitted light.

Fly ash is formed at high temperatures during the combustion of pulverized coal. At these high temperatures, the ash forming constituents of the coal melt, fuse, and form spherical masses, in most cases (Figure 3).

The spherical particles containing iron oxide are essentially magnetic (Figure 4) and can be removed by magnetic processing.

Residues from the magnetic separation are essentially devoid of spherical iron bearing particles (Figure 5); however, a fair amount of iron oxide remains as ragged, irregularly-shaped particles, some of which have a cellular appearance (Figure 6). This material is believed to be thermally decomposed pyrite which had originally replaced fusain in the coal. This pyrite was thermally decomposed by the combustion process but did not fuse into the spherical mass which is typical of fly ash particles. This material may not be magnetic.

Examination of the magnetically separated iron oxide shows a great abundance of spherical particles, all of which are not pure iron oxide. Some of this material is the previously mentioned "silicated iron oxide". This silicated material appears to exist in two forms, one in which the magnetic iron oxide occurs as discrete particles imbedded in a spherical mass of silica (Figure 7), the other as a solid core of magnetic iron oxide surrounded by a siliceous mass (Figure 8).

While this material can be readily separated from the fly ash by magnetic methods, the associated siliceous material is also removed. This unfortunately is the major problem encountered in upgrading the iron oxide.

Future work on this project will be concerned with development of improved crushing and separation methods for upgrading the iron oxide to meet specifications for new, as well as existing, process applications.

## Summary

Upgrading iron oxide derived from fly ash is complicated by the physical state of the iron as it occurs in the fly ash. The "silicated" state of the iron also presents problems in analysis. Ordinary crushing of the material to release the iron embedded in the silica has not been found to be an effective means to upgrade the iron oxide. The silica content remains unchanged and the magnetic content decreases. Further experimental work is necessary to achieve the desired degree of upgrading.

## Acknowledgement

The photomicrographs were taken by Richard Moses, BCR petrographer.

TABLE 1. COMPARATIVE DATA ON FLY ASH-DERIVED IRON OXIDE AND COMMERCIAL MAGNETITE

Screen Analysis - Percent Retained

| Screen Size | Iron Oxide | Commercial Magnetite Grade 2 |
|---|---|---|
| + 50 mesh | 0.15 | 3.8 |
| 50 x 70 mesh | 0.25 | 4.4 |
| 70 x 100 mesh | 0.70 | 4.4 |
| 100 x 140 mesh | 0.35 | 6.2 |
| 140 x 200 mesh | 6.35 | 7.6 |
| 200 x 325 mesh | 26.80 | 13.5 |
| - 325 mesh | 62.40 | 64.5 |

Approximate Chemical Analysis, Percent by Weight

| | Iron Oxide | Commercial Magnetite |
|---|---|---|
| Iron | 57 | 62 |
| Silicon | 6 | 2 |
| Manganese | 0.8 | 0.2 |
| Aluminum | 3 | 4 |
| Calcium | 0.2 | 0.50 |
| Magnesium | 0.04 | 1.50 |
| Moisture | 0.10 | 0.50 |

Physical Properties

| | | |
|---|---|---|
| Specific gravity | 3.82 | 4.80 |
| Magnetics | 90% | 94% |

TABLE 2. COMPARISON OF RESULTS OF IRON ANALYSES USING VARIOUS METHODS

| Sample | Sodium Carbonate Fusion * % | Caustic Fusion Initial % | Caustic Fusion Secondary % | Hydrochloric Acid Extraction % | Hydrofluoric Acid Extraction % |
|---|---|---|---|---|---|
| 1 | 5.9 | 8.7 | - | 6.0 | 11.6 |
| 2 | 14.9 | 11.0 | 14.9 | 3.2 | 18.4 |
| 3 | 4.5 | 5.7 | - | 2.7 | 8.0 |
| 4 | 4.6 | 8.1 | - | 7.2 | 11.1 |
| 5 | 36.8 | 36.7 | 4.5 | 41.3 | 44.1 |

* Results reported by another laboratory.

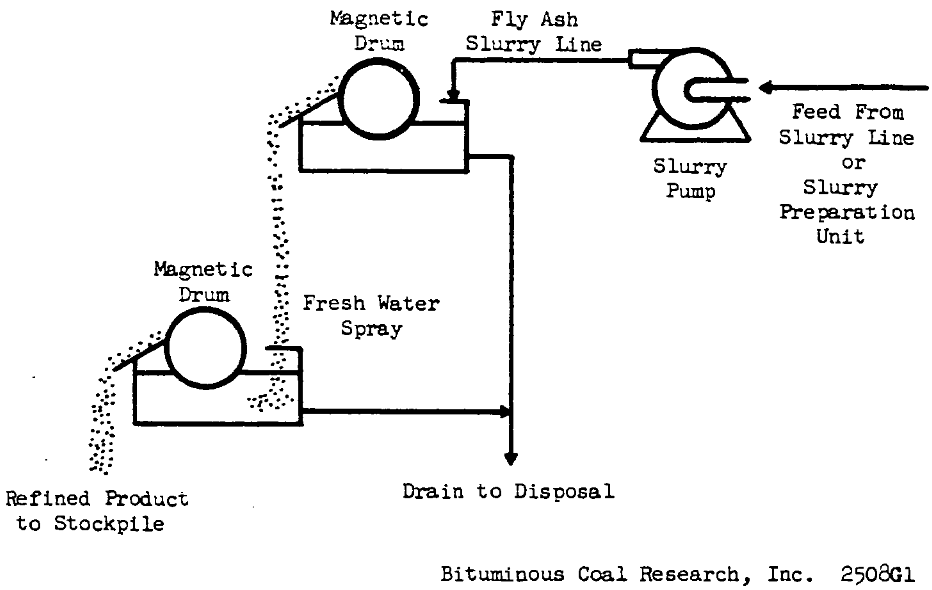


Figure 1. Wet Magnetic Separation Process for Recovering Iron Oxide From Fly Ash

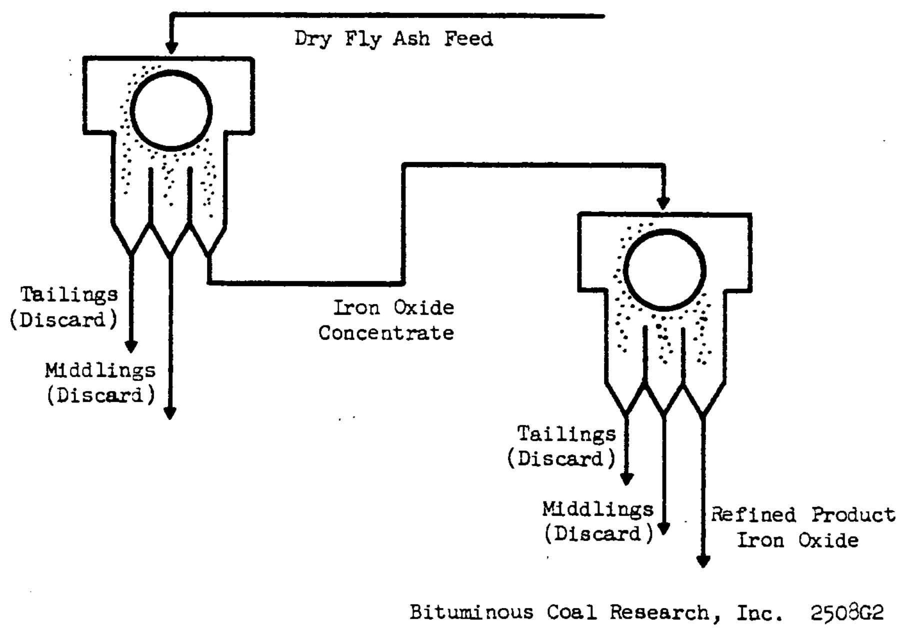


Figure 2. Dry Magnetic Separation Process for Recovering Iron Oxide From Fly Ash

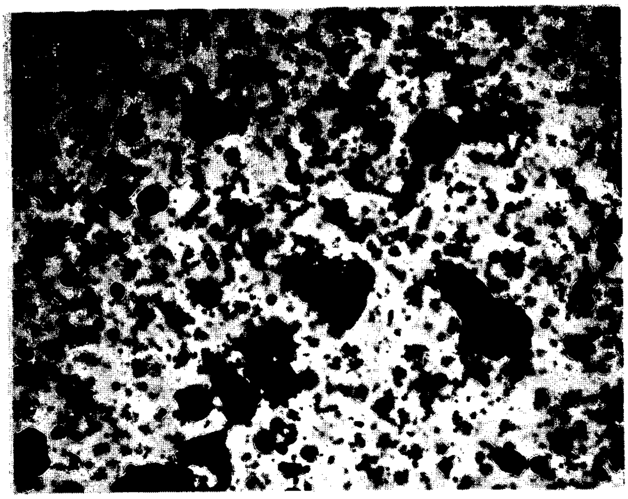

Figure 3. 2508P1
Fly Ash from Mechanical Dust Collector of an Operating Power Station Burning Pulverized Coal (200x)

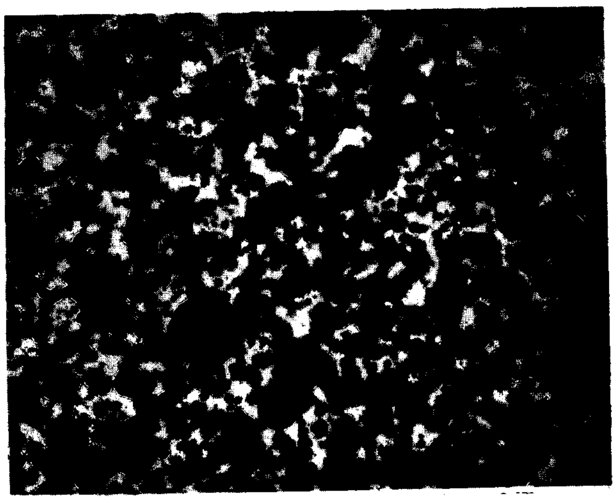

Figure 4. 2508P2
Iron Oxide Magnetically Separated from Fly Ash (200x)

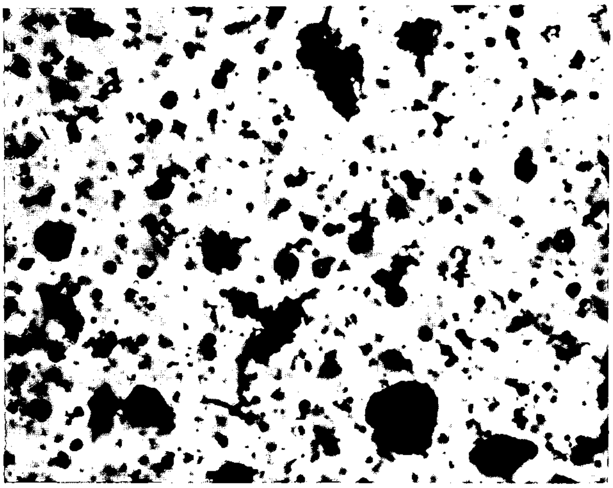

Figure 5. 2508P3
Non-Magnetic Residue from Magnetic Separation of Fly Ash (200x)

Figure 6. 2508P4
Unfused Iron Oxide (top center) in Non-Magnetic Fraction of Fly Ash (400x)

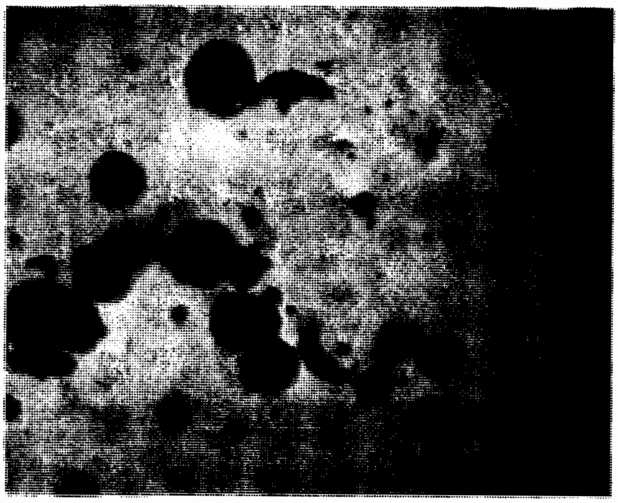

Figure 7. 2508P5
Discrete Iron Particles Enclosed in Siliceous Mass Found in Magnetic Fraction of Fly Ash (200x)

Figure 8. 2508P6
Iron Oxide Sphere Enclosed by Siliceous Mass Found in Magnetic Fraction of Fly Ash (400x)